Four BNLers Win Environmental Stewardship Awards

April 22, 2005

At the annual Earth Day Awards Ceremony held on April 22 by George Goode, Manager of the Environmental and Waste Management Division, Goode and Peter Bond, Interim Deputy Director for Science & Technology, presented four BNL employees with Environmental Stewardship Awards for demonstrating an outstanding effort in pollution prevention, waste minimization, or conservation. The four were: Mary Ann Corwin, National Synchrotron Light Source Department (NSLS); Michael Kindya, Plant Engineering Division (PE); Michael Paquette, PE; and Joel Scott, Collider-Accelerator Department (C-AD).

George Goode (left) and Peter Bond (right) present the awards to (from left) Joel Scott, Mary Ann Corwin, Michael Paquette, and Michael Kindya.

Mary Anne Corwin, who heads the User Administration Office at the National Synchrotron Light Source (NSLS), was awarded for leading the effort to create a virtually "paperless office." By using the Lab's web-based Guest Information System and the NSLS Proposal, Access, Safety and Scheduling (PASS) System, considerable resources were saved by reducing the use of paper copies, improving records retention methods, and recovering office space.

— Liz Seubert

NSLS Daughters and Sons are Forensic Scientists for a Day

April 28, 2005

On April 28, approximately 30 daughters and sons of NSLS staff and scientists questioned suspects, analyzed crime-scene evidence, and caught a thief — all in one morning. The activities were part of the national Take our Daughters and Sons to Work Day. This year at the NSLS, the day had a theme: forensic science.

First, the children gathered in the NSLS seminar room to hear a brief safety talk by Nick Gmur, and then learned about light in its different forms from NSLS scientist Lisa Miller, in her talk, "What Kind of Light?" The kids guessed which type of light is used in many common objects, such as remote controls and microwave ovens.

NSLS scientist Randy Smith demonstrates the process of "blood"-typing.

After the talk, the main event began. Lisa introduced the crime — The Case of the Missing iPod — and then presented the "evidence" that had been found at the scene: a white powdery substance, a black powdery substance, a strand of hair, and a piece of notebook paper. In a nearby trash basket, there was also a soda can with a drop of "blood" on it.

The "victim," NSLS student researcher Meghan Ruppel, then told her story. She said she was studying for a test in the library, got up to talk to some friends, and returned to find her iPod missing. Her story revealed several possible suspects: Adele Wang, Meghan's friend; Michael Appel, the library's custodian; Laura Mgrdichian, the librarian; and Steve Giordano, a library patron. The suspects filed in, told their own stories of what happened, and were questioned by several of the children.

After seeing the evidence, hearing the victim's and suspects' stories, and interrogating everyone involved, the daughters and sons split into groups to analyze the evidence. They tested the "blood" on the soda can to determine the blood type, and dusted the can and the victim's notebook paper for fingerprints. They compared their results with the suspects' blood types and fingerprints.

Next, the children went down to the NSLS experimental floor. There, NSLS scientists Tony Lanzirotti and Bill Rao helped them analyze hair samples from the suspects using x-rays,

Participants in the NSLS Sons and Daughters Day which measure the levels of various trace elements (such as zinc, copper, or calcium) that are present in the hair. The analysis yielded a unique "signature" for each sample, which was compared to the signature of the strand of hair found at the crime scene. The group also analyzed the powders found at the crime scene with a synchrotron infrared microscope. They determined that the white powder was powdered sugar and the black powder was ground coffee.

With all the evidence properly analyzed, the daughters and sons returned to the seminar room, knowing who had taken the iPod.

And who was the culprit? Laura the librarian! She took the iPod "because iPods are not allowed in the library." In her introductory story, she only revealed that she made coffee and ate a powdered doughnut that morning. Caught, however, she further explained that she noticed a soda can on Meghan's desk and, because food and drinks are not allowed in the library, went over to throw it way. She cut her finger on the top, leaving a blood drop. Then she noticed the iPod, another forbidden item. She took it, but left behind coffee and sugar powders, a strand of hair, and many fingerprints. The NSLS daughters and sons successfully analyzed these clues to catch the true "thief."

NSLS scientist Lisa Miller uses a synchrotron infrared microscope to identify the unknown white and black powders.

— Laura Mgrdichian

Women in Science Career Day at BNL

May 9, 2005

Ten female students from the Henry Viscardi School, an Albertson, Long Island-based school for children with physical disabilities and health impairments, learned about scientific careers first-hand from female researchers at BNL. The career day was coordinated by Brookhaven Women in Science and funded by Brookhaven Science Associates. During the day, Jeanne Petschauer (standing, left) from the Community Relations Office gave an overview of BNL's breadth of scientific interests, and Lisa Miller (right) of the NSLS discussed her path to becoming a chemist and her research on such diseases as osteoarthritis, osteoporosis, and Alzheimer's disease. Linda Bowerman of the Atmospheric Sciences Division explained some of BNL's environmental research, and physicist Angelika Drees described her work with other scientists to discover more about the earliest moments of the universe now being studied at the Relativistic Heavy Ion Collider. Among the students present were (front, from left) Adeline Joshua and Stephanie Avramenko.

— Diane Greenberg

BNL Workshop on Intense Coherent THz Pulses

May 10, 2005

The first workshop on intense coherent THz pulses at Brookhaven National Laboratory (BNL) was held during the spring of 2005. Participants included scientists from regional universities and institutions as well as members of the BNL scientific community.

The workshop focused on the science enabled by linac sources of intense coherent terahertz (THz) radiation. The NSLS Source Development Lab (SDL) linac is one such source, producing 100-microjoule, single-cycle coherent pulses with spectral content reaching to a few THz. The full-day workshop began with a presentation by Xijie Wang (NSLS) that described how the SDL linac produces ultra-short, high-charge electron bunches that generate coherent THz pulses or deep ultraviolet free-electron laser light. Larry Carr (NSLS) followed with a description of the SDL's THz pulses, including measurements of the pulse energy, spectral content, and electro-optic detection instrumentation. He emphasized applications where the

Participants in the Intense Coherent THz Pulses workshop

THz pulses serve as an excitation source rather than simply a probe of a material's response. Tony Heinz (Columbia University) gave an overview of THz methods, current scientific efforts, and the frontiers opened by a source of high intensity THz pulses. Ivan Bozovic (BNL Materials Science) described ultrafast THz studies of correlated electron systems and complex oxides — material systems of great interest at Brookhaven. Toni Taylor (Los Alamos National Laboratory) presented other THz studies of complex materials in a seminar the following day. The National Institute of Standards and Technology (NIST) uses THz methods to study complex biomolecules. Ted Heilweil (NIST) discussed that activity as well as applications of THz in homeland security and imaging. It has been noted that the magnetic field portion of a THz wave could be sufficient to change the magnetization state of some magnetic alloys, a topic Dario Arena (NSLS) summarized in his talk. The connection between THz and magnetization dynamics was also described in Toni Taylor's presentation.

Attendees were given a tour of the SDL facility and the location for future THz studies. This was followed by a discussion session that helped to define experimental directions and provide recommendations on the facility and instrumentation requirements.

For more details, see http://www.nsls.bnl.gov/newsroom/events/workshops/THz/. This workshop was sponsored by the NSLS.

— Larry Carr

A Passion for Synchrotron Science and its Future: News from the 2005 NSLS Annual Users' Meeting

May 23-25, 2005

The speakers at the main session of the 2005 National Synchrotron Light Source (NSLS) Annual Users' Meeting, held on Tuesday, May 24, at Brookhaven National Laboratory (BNL), spoke on many different topics. But they all conveyed fierce enthusiasm for the science performed at the NSLS and expressed hope that its proposed successor, the world-leading NSLS-II, would become a reality.

NSLS Users' Executive Committee Vice-Chair Peter Stephens welcomed the audience to the main meeting, setting a positive and enthusiastic tone for the day's events. He then opened the stage to BNL Director Praveen Chaudhari.

Chaudhari commended many attendees for their work to advance NSLS-II. "You've helped tell us what is needed in this new light source, including the workshop last fall that defined NSLS-II," he said. "The struggle to get NSLS-II is just beginning — we need to design it and then get funding. But once the machine is built, you'll have the best machine in the world, and we need your help to make that happen."

Pat Dehmer (third from right), head of the Office of Basic Energy Sciences within the Department of Energy's Office of Science, was a special guest at the 2005 NSLS Annual Users' Meeting. She stands with (from left) NSLS Associate Chair for User Science Chi-Chang Kao, incoming Users' Executive Committee (UEC) Chair Peter Stephens, NSLS Chairman and Associate Lab Director for Light Sources Steve Dierker, BNL Director Praveen Chaudhari, and outgoing UEC Chair Larry Shapiro.

Chaudhari then introduced Patricia Dehmer, head of the Office of Basic Energy Sciences within the U.S. Department of Energy's Office of Science, who plays an important role in efforts to move NSLS-II forward. Dehmer elaborated further on the status of the proposed new facility and made several key points, many concerning the tough budget years ahead.

The Office of Science, she said, led by Raymond Orbach, has set a philosophy in place for fiscal year 2006: making the U.S. the leader in every major field of research, regardless of the declining budgets to come. "These are very scary times, and being bold and aggressive is probably the only way to face this," she said.

However, her talk was full of encouraging messages. NSLS-II, she said, falls into one of the "mission challenges" of the Office of Basic Energy Sciences — that is, enabling the construction of major scientific facilities.

"NSLS-II will undoubtedly be the world's finest synchrotron; it will be a stunning facility," she said. Soon, she added, she and Orbach will present the Laboratory's NSLS-II proposal to Deputy Secretary of Energy Clay Sell for his approval.

She spoke emphatically to NSLS-II supporters. "I think we have a very high probability for success with NSLS-II. But we need your help, too. You have to understand the realities of the budget and be sophisticated when you talk to folks in Washington. You can't rely on Congress to launch something like NSLS-II — you have to talk to the administration."

In her closing remarks, Dehmer left off on a very positive note. "This laboratory has a wonderful history of constructing and operating major user facilities," she said. "The run of the NSLS has been nothing short of remarkable, and NSLS-II will take that tradition and move it into the future." She also praised

NSLS Chairman Steve Dierker. “Steve has been a superb leader for NSLS-II, and there’s no way we could have made our case to Ray Orbach without him. NSLS-II has moved up in the DOE 20-year plan largely because of Steve’s efforts.”

Dierker, who spoke next, showed the audience that the NSLS continues to thrive, even as third-generation synchrotrons draw more and more users. “We’ve held our own and then some,” he said. “The number of users served by the NSLS has been stable at about 2,300 per year.”

He described the NSLS as “very cost effective, highly productive, and highly reliable.” Since 2001, the facility has met many key goals, such as maintaining and strengthening its user program, expanding its user base, and developing a compelling proposal for NSLS-II.

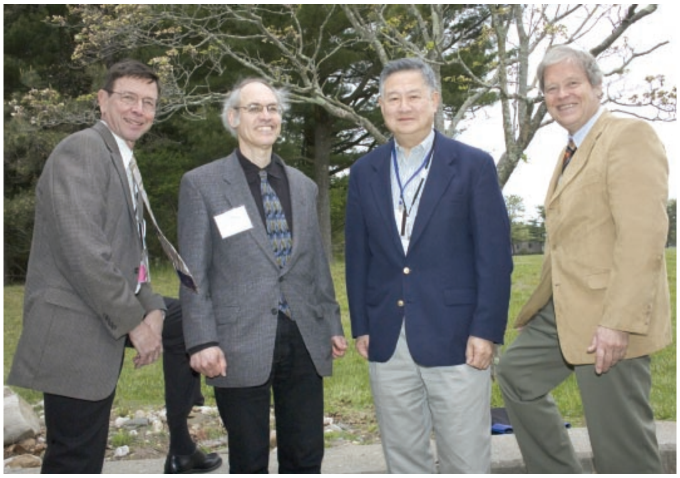
In addition, there has been a “dramatic” evolution of NSLS beamlines, including better support to several beamlines to make them more useful and modern, and many major beamline upgrades.

Looking into the future, Dierker said he looks forward to continuing user input on NSLS-II. “The community has responded very enthusiastically and vigorously,” he said. “I think we have put forward a compelling design that is critically needed in order to probe materials at high-energy resolution, and at spatial resolutions on the order of one nanometer, which would be unprecedented.”

“There is a host of important and exciting scientific opportunities that will be enabled by NSLS-II,” he concluded. “This is something the U.S. absolutely needs to regain leadership in synchrotron radiation science.”

Next, in the first scientific talk of the day, Henk Schenk of the University of Amsterdam discussed “The Structure of Cocoa Butter and the Quality of Chocolate.” In this interesting presentation, Schenk described his group’s work using x-ray diffraction to study the structure of cocoa butter. Cocoa butter is an essential component of chocolate that determines the

The 2005 NSLS Annual Users’ Meeting Planning Committee: (from left) Mary Anne Corwin, Liz Flynn, Melissa Abramowitz, Ron Pindak, Lisa Miller, Dan Fischer, Gretchen Cisco, and Peter Stephens.



Speakers at the main meeting included (from left) Bob Casey (BNL-NSLS), John Rehr (University of Washington), Benjamin Chu (Stony Brook University), and Henk Schenk (University of Amsterdam).

chocolate’s characteristic properties, such as its sheen and meltability. By studying the various phases of cocoa butter via melting-cooling processes, he and his group patented a method to produce chocolate that stays fresh longer than other chocolates, and even devised a chocolate-making machine.

Schenk was followed by a talk on safety delivered by Peter Stephens and Bob Casey, the NSLS Associate Chair for Environment, Safety, Health, and Quality. In a back-and-forth style, Casey and Stephens discussed safety from the point of view of NSLS users and administration, particularly in the wake of the electrical incident last year at the Stanford Linear Accelerator (SLAC). The issues raised during their talk were presented in further detail, and subject to more extensive discussion, at a special “Electrical Safety in the Research Community” workshop the following day.

The workshop covered several topics. NSLS Safety Officer Andrew Ackerman discussed National Fire Protection Association electrical standards implemented at the NSLS. He also elaborated on a new NSLS rule that all electrical devices in the NSLS be certified by a nationally recognized testing laboratory within five years, including equipment brought in by users. He showed several photos of unsafe and/or “homemade” electrical equipment and configurations found on the NSLS floor, which illustrated the need for such rules.

Bob Chmiel, the NSLS Environmental, Safety, and Health engineer, expanded on this. He displayed actual examples of unsafe electrical configurations found during routine NSLS inspections, and encouraged users to routinely check their equipment. Finally, Casey gave a more detailed account of the SLAC incident, the many violations of procedure and practice that led to it, and lessons learned. He also went over some recent NSLS electrical incidents, and the lessons learned from them.

The second scientific talk at the main meeting was delivered by John Rehr of the University of Washington. Rehr spoke

This year's poster session winners were (from left) Tejas Telivala (Stony Brook University), Ashtosh Ganjoo (Lehigh University), Angelo Dragone (BNL-Instrumentation Division), Holger Fleckenstein (SBU), Meghan Ruppel (SBU), and Brandon Chapman (BNL-NSLS).

about the theory involved in interpreting x-ray data obtained from many synchrotron analysis techniques, such as extended x-ray absorption fine structure (EXAFS) and nuclear resonant inelastic x-ray scattering.

In the afternoon, Benjamin Chu from Stony Brook University talked about the polymer experiments he performs with his group at beamline X27C, using wide-angle x-ray diffraction (WAXD) and small-angle x-ray scattering (SAXS). Their end station contains several specialized instruments, such as spinning, stretching, and high-pressure devices, which allow them to investigate various properties of the polymers.

Steve Almo

Next, Steve Almo from the Albert Einstein College of Medicine discussed "Structural Genomics in the 3rd Millenium." Almo said that scientists are solving protein structures at amazing rates, but that the future of structural biology is determining the structures of protein complexes — many proteins interacting at once. He described a new technique to study proteins, called synchrotron x-ray footprinting, which may help structural biologists look beneath cell membranes to study many cell components at once.

Peter Abbamonte

Finally, NSLS scientist Peter Abbamonte presented his work on antiferromagnetism, a state of magnetism in certain materials in which ions orient themselves into regions of opposite alignment, called "stripes." Antiferromagnetic materials can become superconductors, and Abbamonte and his group are trying to determine if stripes play a role — do they assist or compete with superconductivity?

At the end of Tuesday's main meeting, the outgoing NSLS Users' Executive Committee Chair, Larry Shapiro, announced the three newest members of the UEC: Chris Jacobsen of Stony Brook University (SBU), Steve Almo, and Chris Cahill of George Washington University. NSLS scientist Lisa Miller, the poster session organizer, announced the poster sessions winners: Brandon Chapman (BNL-NSLS), Angelo Dragone (BNL-Instrumentation), Holger Fleckenstein (SBU), Ashtosh Ganjoo (Lehigh University), Meghan Ruppel (SBU), and Tejas Telivala (SBU).

Later that day, meeting participants attended the evening banquet in Berkner Hall for good food, drinks, and conversation. During dinner, photos of the NSLS and NSLS staff cycled on a large screen at the front of the room, sparking conversations. Stephens also presented the UEC Community Service Award to Tony Lenhard.

After dinner, NSLS historian Robert Crease treated everyone to a bit of history during a special presentation. In a narrative accompanied by old photos, he recounted the days before the NSLS was built, the roadblocks encountered before and during its construction, and the ultimate success of the facility.

NSLS historian Bob Crease took the banquet attendees back in time during his presentation on the history of the NSLS.

During the two days surrounding the main meeting, Monday the 23rd and Wednesday the 25th, several additional workshops were held at locations across the Laboratory. They were "Nanomagnetism: Materials and Probes," "Imaging Nanoscale Structure in Biominerals: New Results and Challenges," "The Impact of Cryogenic Specimen Automounters on the Future of Macromolecular Crystallography," "Spectroscopic Studies of Nanoscaled Systems," "Application of Small-Angle X-Ray Scattering to Biological Structures," and "In-situ Analyses in Environmental and Chemical Systems."

— Laura Mgrdichian

Nanomagnetism: Materials and Probes Workshop

May 23, 2005

The connection between magnetism and nanoscience is clear: The nanometer is the natural length scale of magnetism, as it characterizes the domain wall. Unanticipated phenomena occur when the materials' structural scale coincides with the magnetic length scale. To support research into nanomagnetism, recent advances in materials synthesis and nanofabrication technology have made it possible to create a wide range of nanomagnetic systems with unprecedented precision. These novel magnetic systems are used as model systems for testing longstanding theories in magnetism, as well as for exploring new device concepts and applications. In parallel, synchrotron radiation, with its unique polarization properties, tunability, and time structure, has become an indispensable tool for the study of magnetism and magnetic materials.

The 2005 NSLS Users' Meeting Workshop "Nanomagnetism: Materials and Probes" provided an overview of the latest synchrotron characterization techniques as well as introduced new materials concepts for magnetic materials. The workshop was

Nanomagnetism: Materials and Probes workshop attendees

jointly sponsored by the Brookhaven National Laboratory Center for Functional Nanomaterials, a new U.S. Department of Energy nanoscience user facility. Presentations were delivered by seven experts in the application of synchrotron radiation to magnetic materials, and in the synthesis and application of magnetic nanomaterials, with the goal of presenting an up-to-date snapshot of the forefront issues in nanomagnetism.

Daniel Haskel of the Advanced Photon Source at Argonne National Laboratory inaugurated the workshop with a presentation on recent synchrotron characterization advances that elucidate the role surfaces and interfaces at play in the overall magnetic response, especially in thin-film layered systems.

Professor J.M.D. Coey of Trinity College in Dublin, Ireland, followed with surprising but well-documented results that systems with nominally no d-electrons, such as HfO_2 and RhO_2, exhibit ferromagnetism under certain conditions. The results are currently attributed to the defect state of the material.

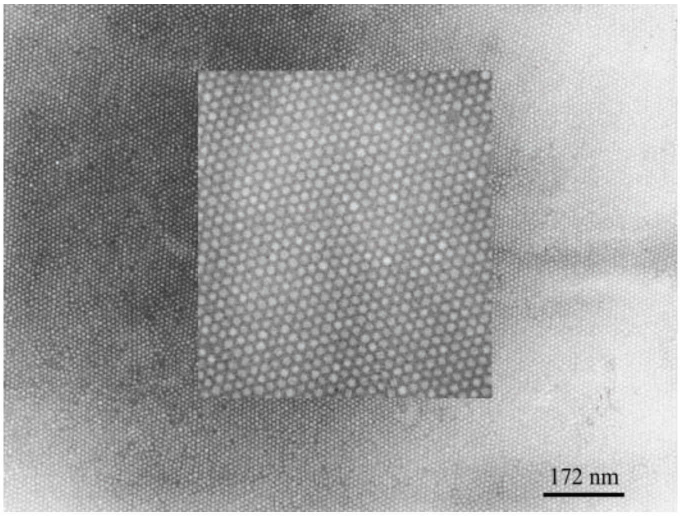


Figure 1. Large view (1.5 mm x 1.5 mm) of uniform ε-Co ferromagnetic nanoparticles with a high degree of order induced by lateral com-pression to form a compressed Langmuir film. The inset shows the presence of dislocations but no grain boundaries in the film. (D. Farrell, Y. Cheng, R.W. McCallum, M. Sachan, and S.A. Majetich, *J. Phys. Chem. B*, **109(28)**, 13409-13419 (2005).

Continuing the focus on novel magnetic materials, Distinguished Professor Myriam P. Sarachik of City College–CUNY presented new results on molecular magnets or single-molecule magnets. These systems, which bridge the classical and the quantum worlds, hold potential as quantum computation materials (qubits) and exhibit clear quantum-mechanical tunneling signals under the influence of magnetic fields.

Professor Sara A. Majetich of Carnegie Mellon University discussed recent results obtained from highly uniform monodisperse arrays of ferromagnetic Fe, Co, and FePt nanoparticles (**Figure 1**). The competition between dipolar and anisotropy energies in these systems creates spin-glass-like mictomagnetic behavior, as revealed by small-angle x-ray scattering and small-angle neutron scattering.

Glenn A. Held of the IBM T. J. Watson Research Center in Yorktown Heights, New York, presented an overview of magnetic ferrite nanoparticles encapsulated by biologically active molecules to create bio-functionalized entities for monitoring and influencing cellular processes.

The workshop concluded with two presentations on advanced synchrotron techniques for magnetism characterization. Dario Arena of the NSLS discussed new techniques that probe the dynamics of magnetization precession in the time domain, with element specificity. In this work, time-resolved magnetic circular dichroism in a pump-probe architecture was employed to examine the moment response in permalloy-based systems. Andreas Scholl of Lawrence Berkeley National Laboratory described the application of ultrafast x-ray pulses (picosecond to femtosecond) to image magnetic dynamics with high spatial resolution. As an example of the technique, real-time movies with associated analyses of the precessional dynamics of magnetic vortices were presented.

— Laura H. Lewis and Chi-Chang Kao

Synchrotron Imaging of Biominerals Workshop

May 23, 2005

Biominerals, the mineralized tissues of animals, plants, and microorganisms, have inspired humanity since prehistory. Bones and shells have been used for tools, currency, symbolic objects, and art in every culture. Both the fascination and the importance that biominerals present for science are made clear in Darwin's 19th century writings, and in D'Arcy Wentworth Thompson's 1917 *On Growth and Form*, with its memorable cover illustration of the multi-chambered calcium carbonate nautilus shell.

Now, biomineralization is a field of study in its own right. Biologists, paleontologists, materials chemists, physicists, engineers, and medical professionals all contribute to our understanding of how biominerals grow; how they achieve their submicron hierarchical architectures and their precise control over crystal orientation and habit; how they are able to stabilize non-thermodynamically-favored mineral polytypes; and most importantly, how the biomineralization process might be harnessed or mimicked to produce new nanostructured, multicomponent materials for medicine and technology.

Synchrotron Imaging of Biominerals workshop attendees

Recent advances in synchrotron science, as applied to these materials, have uncovered a wealth of new information in the past ten years. Synchrotrons now enable diffraction-enhanced imaging, x-ray microbeam analysis, computed tomography, and phase radiography to probe the microstructures of biominerals. Chemical information is obtained from soft x-ray photoemission and infrared spectromicroscopy techniques, with submicron spatial resolution. High-resolution diffraction, small-angle scattering, and x-ray absorption methods all contribute to the picture of the crystalline and amorphous phases formed. Finally, many of these experiments are sensitive to crucial organic components: the matrix of proteins and polysaccharides that help give biominerals their special properties.

Such new results and challenges were highlighted in a workshop, "Synchrotron Imaging of Biominerals," at the 2005 Annual NSLS Users' Meeting. Our six invited speakers were followed by four short talks from BNL and Stony Brook University scientists, including student presentations. This workshop was also videotaped and is posted on the web at *http://www.solids.bnl.gov/~dimasi/nsls05ws2/*.

Matthias Epple

Matthias Epple of University Duisburg-Essen, Germany, emphasized the importance of studying well-characterized biological samples in collaboration with biologists, who can help interpret the information. Epple then went on to show how high-resolution powder diffraction, EXAFS, and tomography were used in tandem to obtain structural information from a variety of small animal shells and structural organs. High-resolution measurements are necessary to distinguish between calcite, magnesian calcite, and small amounts of metastable polytypes only observable with synchrotron radiation. In more unusual animals, extremely unlikely minerals, such as calcium sulfate hemihydrate statoliths in deep-sea medusae, were discovered. Finally, selected amorphous calcite carbonate mineral formers were surveyed. Effective measurement of the metastable amorphous biominerals is particularly valuable since biomineralization is thought to often proceed by means of amorphous precursors.

Emil Zolotoyabko

Emil Zolotoyabko, from the Technion-Israel Institute of Technology, focused on his newly developed technique for depth-resolved measurements using energy-variable x-ray diffraction. The motivation is that biominerals have complex, multilayered structures. While many synchrotrons have developed micron beam spots to resolve diffraction patterns laterally (across the sample surface), it is necessary to match this spatial resolution in the third direction for a complete picture. Zolotoyabko's technique is based on theoretical analysis of the shapes of diffraction profiles taken under slight misalignment of the diffraction instrument. The interplay between the probability of the x-ray registration in the detecting system and the depth-dependent attenuation of the primary x-ray beam defines at each energy a specific depth from which the maximum diffraction signal is collected. By measuring diffraction profiles at different energies, the depth-dependent effects in the preferred orientation, grain size, microstrain fluctuations, and residual strains could be observed. Zolotoyabko highlighted information obtained from mollusk shells in which microstructural parameters in the successive layers of a material can tell the story of a biomineral growth.

Stuart R. Stock of Northwestern University presented the history and latest advances in x-ray absorption computed microtomography and phase radiography. These non-invasive three-dimensional imaging techniques can be applied to both medical and materials studies. Stock first gave a detailed discussion of the technique's mathematical basis and practical limitations. He next described how spatial and temporal variations in microstructure could be observed for mouse skull tissue response to bone resorption-inducing agents, for the mineralized collagen of regenerated newt limbs, and to study a rabbit model for vascular mineralization, probing treatments to ameliorate the effects of cholesterol. Finally, interesting questions about some similar proteins implicated in skeletal evolution were brought up, contrasting the calcium carbonate skeletons of echinoderms with the calcium phosphate bones of vertebrates: How does the spatial distribution of protein relate to the microarchitecture and mineral density? Sea-urchin teeth exhibit a remarkable array of materials engineering "tricks" in the design of their composite microstructures, as a *functional* analysis of the images demonstrates.

Stuart R. Stock

Gelsomina "pupa" De Stasio, University of Wisconsin - Madison, and Synchrotron Radiation Center. A lively talk from De Stasio showcased SPHINX, an x-ray PEEM spectromicroscope. With monochromatic soft x-rays incident onto the sample surface, photoelectron emission imaging with a field of view of 2 to 200 microns can be accomplished. By scanning the energy, XANES or EXAFS spectra are obtained, with sensitivity to all elements present in biological and mineral systems. This makes chemical and spectroscopic information available with 10 nm spatial resolution. De Stasio discussed wide-ranging examples of biomineralization not previously covered, including the formation of bacterial biofilms and the "reverse-biomineralization" activity of antifreeze proteins — emphasizing, in all cases, the organic-inorganic interface. She also presented another mystery in mollusk nacre: a polarization dependence, suggesting that the carbonate groups in aragonite do not align along the tablet axis as is currently thought.

Gelsomina "pupa" De Stasio

Helga Lichtenegger, from the Vienna University of Technology, presented research on another unusual family of biominerals: marine worms whose mandibles are reinforced with the copper-based mineral atacamite. Lichtenegger has explored the distribution of copper, zinc and iron in *Glycera* and *Nereis* worm jaws, and the experimental achievement is an elegant combination of microbeam x-ray absorption spectroscopy, diffraction, and small-angle scattering. Each of these techniques had a necessary role in determining which metals form crystals within the jaws, and which instead are present in trace quantities and may play some role in the tissue other than structural support; and in the case of SAXS, what morphology the mineralized parts exhibit and how that relates to the structure of the whole jaw.

Helga Lichtenegger

Teresa Nicolson joined us from the Oregon Hearing Research Center and Vollum Institute, Oregon Health and Science University. Nicolson studies the molecular basis of mechanotransduction in sensory hair cells. This research uncovered a puzzle in biomineralization when genes required for hearing and balance in zebrafish were explored. Nicolson demonstrated that a previously unknown gene, *starmaker*, was required for the proper development of the calcium carbonate otoliths or stone-like particles, which serve as balance sensing organs in the ear. The *starmaker* activity was experimentally disrupted in zebrafish embryos. With mild disruption the aragonite mineral otoliths were changed from round to chunky morphologies, and in more excessive cases another calcium carbonate mineral, calcite, was formed. Synchrotron microbeam diffraction was necessary to detect these changes.

Teresa Nicolson

Selected local efforts were showcased in short talks following the invited program. Zhong Zhong (NSLS) presented the superior contrast achieved from Diffraction Enhanced Imaging using x-rays. Meghan Ruppel (NSLS) used infrared spectromicroscopy to compare the chemical makeup of microdamaged versus undamaged bone. Karthikeyan Subburaman (Stony Brook University) described the biomimetic mineralization of synthetic protein films, and Seo-Young Kwak (NSLS) measured the orientation and Mg-incorporation of calcite grown on functionalized self-assembled monolayers.

Interdisciplinary symposia can be a challenge as well as a pleasure for the participants. Aside from the compliments, other

Biomineral Imaging at the NSLS

comments (that the organizer, perhaps, was not intended to hear) included: “That was way too technical for me”; “The biology was over my head”; and “Definitely not the way I’m used to thinking about problems.” It’s our hope that such symposia can continue to increase awareness of the problems and methods encountered in a research area like biomineralization, which presents so many faces. For this reason, we are especially appreciative of the support and participation that this workshop received. We would like to thank the NSLS, the NSLS Users’ Executive Committee, the Center for Functional Nanomaterials, and BNL videographer Alex Reben.

— Elaine DiMasi

The Impact of Cryogenic Specimen Automounters on the Future of Macromolecular Crystallography Workshop

May 23, 2005

Cryogenic “automounters” are becoming common at synchrotron x-ray sources and in a few commercial laboratories, employed especially in macromolecular crystallography (PX). There are several commercially available models, and several home-built ones, used in a range of synchrotron radiation sources around the world. This workshop gave the PX community an opportunity to hear the major builders of these instruments describe important features of the operation of their instruments, and the ways the scientific community is using them.

Thomas Earnest’s group at Lawrence Berkeley National Laboratory’s Advanced Light Source (LBL-ALS) designed one of the most imitated automounter systems, based on pneumatic actuators. He summarized their experience at the ALS with the design, now exported to several locations (the NSLS, the Advanced Photon Source (APS), and the Macromolecular Diffraction Facility at the Cornell High-Energy Synchrotron Source (CHESS)). He emphasizes an “open source” view of the design to encourage information sharing and described many small improvements since the original design. They’re working on a Cartesian robot to replace the current system, which would not require the crystal-supply dewar to move, and would have a larger capacity. Dieter Schneider, BNL Biology Department, described his group’s refinement of the LBL-ALS system. They devised a three-jaw gripper that is being adopted at several places, developed innovations that minimize frosting of the lid, and are beginning to develop EPICS-based control. Gerd Rosenbaum, of the Southeast Regional Collaborative Access Team at the APS, described his group’s adaptation of this design. This instrument is just now coming into operation. Quan Hao, CHESS, gave the same sort of report, one innovation being a miniature liquid nitrogen pump for filling the sample dewar, and described his use of a Java-based camera system for sample viewing.

Aina Cohen represented the Stanford Synchrotron Radiation Laboratory (SSRL) robot effort, one of the earliest in the PX community, whose device depends on an industrial robot for flexibility. They have very much experience and have made many refinements to the original system to provide reliability. The SSRL group has developed a web site for sharing ideas about automounters: *http://smb.slac.stanford.edu/robosync/*.

Franck Felisaz, of the Joint Structural Biology Group at the European Synchrotron Radiation Facility’s (ESRF) European Molecular Biology Laboratory (EMBL), Medical Research Council Grenoble, described a robot named SC3 that was developed at the EMBL. Eight of these have been built for ESRF, commercialized through Maatel and marketed through Accel. An especially attractive feature is the software, which can be used with a pocket PC to read bar code labels on crystal-growth trays in order to transfer that information to the data-collection software. Bernard Lavault, also from EMBL, described image-recognition software (“C3D”) for locating and centering crystals on the goniometer. It has an impressive record of finding the real crystal in globs of vitrified liquid. He emphasized the importance of lighting and the ability to vary lighting direction and intensity. Deming Shu, a member of the instrument-development group at the APS, described their design of a custom robotic system based on a commercial six-axis robot. There are many refinements, in particular a force-sensing gripper.

The Impact of Cryogenic Specimen Automounters on the Future of Macromolecular Crystallography Workshop attendees

Several commercially available robotic systems were described as well. Ross Doyle of Mar USA described the MAR Cryogenic Sample Changer, which can be provided as part of MAR dtb (desk-top beamline). This system is used at several places in Europe and at the Structural GenomiX beam line at the APS. Anne Mulichak, a member of the Industrial Macromolecular Crystallography Association Collaborative Access Team (IMCA-CAT) at the APS, described their experience using ACTOR, the six-axis robot produced by Molecular Structure Corporation of The Woodlands, Texas. This instrument is an engineered adaptation of the first such robot ever produced (by Abbott Pharmaceuticals for their internal use). After a few years’ experience at IMCA-CAT the machine is in fairly routine use now. Ehmke Pohl of the Swiss Light Source reported that they purchased one two-Dewar ACTOR system. They put special effort into accurately intersecting the x-ray beam with the crystal, employing x-ray beam position monitors to provide feedback for 10-micrometer positional stability. They're looking into using ultraviolet sensing together with visible light to locate the sample. Finally, Bob He, from the Bruker-Nonius company of Madison, Wisconsin, described yet another quite successful commercial robot based on a six-axis system. This system has found acceptance in the home-lab small-molecule community, and it works just fine in PX.

An hour and a half were reserved at the end of the workshop for open discussion. Both builders and users seriously discussed the standardization of the fixtures the experimenters use to handle their specimens: crystal "caps" and pins, and the cassettes used to carry the caps to the automount robot. The discussion yielded important, detailed ideas that have been passed on to the American Crystallographic Association Data, Standards, and Computing Committee.

— Robert Sweet

Spectroscopic Studies of Nanoscaled Systems Workshop

May 25, 2005

A workshop titled "Spectroscopic Studies of Nanoscaled Systems" was held on May 25, 2005, as part of the National Synchrotron Light Source 2005 Annual Users' Meeting. This workshop was intended to be a forum on the connection between nanoscience and the physics of interacting electron systems, particularly the occurrence of electronic ordering at the nanoscale in oxides, both in extended crystals and in artificial heterostructures. Our hope was that this session might feed the discussion concerning the correlated electron thrust area at Brookhaven Lab's planned Center for Functional Nanomaterials.

The day opened with a presentation on quantum confinement effects in thin films by Tai Chiang from the University of Illinois. Chiang's group carried out angle-resolved photoemission measurements on thin films of lead and determined a direct correlation between the thermal stability of layers of different thickness and the binding energy of the highest occupied orbital. This stability arises from the confinement of the electron in the direction perpendicular to the film and turns out to be periodic in the layer thickness. This periodicity can be thought of as a one-dimensional analogue of the periodic table.

The next speaker was Seamus Davis from Cornell University, who presented scanning tunneling spectroscopy (STS) measurements of the copper-oxide superconductor $Ca_{2-x}Na_xCuO_2Cl_2$. In an earlier study this material was reputed to contain an electronic (i.e. Wigner) crystal, and the current presentation contained additional measurements showing spectral weight modulations at a large binding energy (~ 200 meV). These measurements spurred a continuation of the debate over the definition of the term "Wigner crystal" and what aspect of STS measurements may be thought of as charge order.

After Davis, Ali Yazdani from Princeton University presented similar measurements on underdoped $Bi_2Sr_2CaCu_2O_{8+\delta}$ with trace quantities of zinc. These STS measurements were carried out in the pseudogap state, i.e. $T_c < T < T^*$. Yazdani observed a "checkerboard" pattern that appears to be electronic in origin, disappears when the sample is cooled into the superconducting state, and was speculated to arise from some competing order responsible for the pseudogap. What relation these measurements have to Davis' Wigner crystal, or to the stripe phases seen with neutron scattering, is still a mystery.

After a lunch break, Girsh Blumberg of Bell Laboratories presented Raman measurements of very lightly doped La_2CuO_{4+y} (LCO), as well as the spin ladder material $Sr_{14}Cu_{24}O_{41}$, to characterize the nature of the inhomogeneity in these systems. From his measurements he concludes that the inhomogeneity in LCO is actually two dimensional ("giraffe disorder"), rather than one-dimensional ("zebra disorder").

The discussion turned from intrinsic-occurring order to artificial

Spectroscopic Studies of Nanoscaled Systems Workshop attendees

order with a presentation on quantum wires of underdoped $YBa_2Cu_3O_{6+y}$ by Dale Van Harlingen of the University of Illinois. The Van Harlingen group carried out four-probe transport measurements on such wires and observed quasi-random "telegraph" noise in the IV characteristics. This noise was seen only at temperatures around the pseudogap temperature T^*, and was speculated to arise from stripe domains that exist above T_c and thermally switch at random times. Such switching is not seen in optimally doped wires.

A major topic of discussion at the workshop was whether the physics of oxides differs substantially if patterned into nanostructures. Insight into this subject was provided by Antonio Castro-Neto of Boston University, who gave a theoretical perspective on the edge states that might arise at the surface of a (1,0,0) terminated doped Mott insulator. In a study based on density matrix renormalization group techniques, carried out in collaboration with Stephen White of the University of California at Irvine, Castro-Neto concluded that static stripes form near a terminated surface and, with increasing U, spread out from the bulk and move to the surface. These effects may be important for the properties of transition metal oxide devices.

The session closed with a presentation by Satoshi Okamoto of Columbia University on the subject of electronic reconstruction in oxides with reduced dimensions, such as at surfaces and interfaces in artificial structures. Okamoto carried out a computational study of the interface between $LaTiO_3$ (a Mott insulator) and $SrTiO_3$ (a band insulator), and found that the interface between the two is, in fact, highly metallic and exhibits orbital ordering. The precise form of the orbital ordering depends on the thickness of the interface and the geometry of the structure. Such effects may have already been seen by groups at Bell Labs and in Korea.

— Peter Abbamonte and Peter Johnson

Application of SAXS to Biological Structures Workshop

May 25, 2005

The development new synchrotron sources has enabled challenging small-angle x-ray scattering (SAXS) experiments that require extremely small, yet intense x-ray beams. An example is time-resolved scattering from proteins undergoing folding at millisecond or better time resolution. However, the emergence of these new sources does not make SAXS instruments that are based on older second-generation sources, such as the NSLS, obsolete. Instead, these instruments continue to contribute to biology and biomaterials research. This workshop was intended to show the user community how SAXS can be used to facilitate structural studies of biological systems with examples of studies achievable at second-generation synchrotron sources.

The morning session of this workshop focused on solution scattering from proteins and protein complexes. Dmitri Svergun (European Molecular Biology Laboratory) described applications of the low-resolution structural modeling programs developed in his group. These data analysis methods are based on static measurements that do not require very high source brightness. He gave examples for a broad range of sizes, from individual macromolecules to multi-domain proteins and large macromolecular assemblies. Structural modelling of molecular complexes is particularly effective when combined with high-resolution structures of the constituting subunits.

The scattering at higher angles corresponds to structures at smaller length scales. Lee Makowski (Argonne National Laboratory) showed that scattering within this region contains information on the secondary structures and folding motif of the protein. Wide-angle x-ray scattering (WAXS) data can therefore be utilized to quantify the structural difference between protein structures as a distance in the WAXS space. Proteins known to have similar structural motifs have the shortest distances. This method, therefore, may be a sensitive, global method for detecting structural changes in proteins, narrowly categorizing proteins based on their scattering homology to known folds and elucidating the differences between crystal structures and aqueous conformations.

Scattering from protein solutions is not only capable of charactering the structure of proteins, but also the interaction potential between them. This capability is employed by Annette Tardieu (Centre Nationale de la Recherche Scientifique) to study the conditions that are optimal to obtain protein crystals. The major result from her research is that attraction between protein molecules may be tuned with salt and/or with PEG. The optimal condition depends upon the macromolecular size: With small compact proteins the Hofmeister effect may be sufficient to induce an attractive regime and crystallization, whereas the presence of PEG is required with higher molecular weight complexes.

The last speaker of the morning session was Jack Johnson (The Scripps Research Institute), who studied the maturation of HK97 bacteriophage capsid. This dynamical process proceeds at a moderate speed (on the order of seconds) and was monitored with time-resolved SAXS. This study is a great example of how protein crystallography, electron microscopy, small-angle scattering, and fluorescence complement each other to provide a more complete picture of the biological process that may not be elucidated by any of these techniques alone.

Application of SAXS to Biological Structures Workshop attendees

The afternoon session moved on to the application of SAXS to more complex structures in biological tissues. Due to the presence of various periodic structures, scattering data from tissues often contain diffraction peaks and there is no uniform method of analyzing the data. Ben Hsiao (Stony Brook University) modeled the shape and position of small-angle diffraction peaks from fish bones. His data analysis quantified the diameter of collagen fibrils, the orientation distribution of fibrils, the coherence length, as well as the mineral (calcium phosphate) dimensions and orientation in the bones. Myosins and actin fibers in muscle also produce diffraction peaks. Leepo Yu (National Institutes of Health) studied the structural change in muscles as the chemical-to-mechanical energy conversion that drives muscle movement takes place. In search for materials for replacing defective heart valves, Jun Liao (University of Pittsburgh) studied candidate tissues under biaxial stretch either with constant force or constant displacement. The response of the tissue gives a good indication of its mechanical integrity.

The workshop also included a brief tour of SAXS beamlines X27C and the newly renovated wiggler-based X21 beamline, and ended with a discussion session during which a number of NSLS SAXS users presented their results.

— Lin Yang

In-Situ Kinetic Analyses in Environmental and Chemical Systems Workshop

May 25, 2005

Natural environmental systems, such as soils, sediments, and subsurface aquifers, are always in a state of disequilibrium. Changes in porewater chemistry, biological activity, and chemical properties, such as particle-water interfacial composition, redox potential, and pH, occur over time scales ranging from microseconds to years. Research on the kinetics of chemical and physical processes at different time scales is essential for understanding the fate and transport of environmental contaminants. A workshop organized by Dean Hesterberg and Jeff Fitts for the NSLS Annual Users' Meeting on May 25, 2005, brought together a multidisciplinary group of scientists who discussed in-situ approaches for studying time-dependent chemical processes.

The workshop was dedicated to the memory of the late Dale E. Sayers. Ed Stern (University of Washington) provided a historical perspective on Sayers, discussing how, as a Ph.D. student in 1971, Sayers pioneered (along with Stern and Farrell Lytle) the first correct physical model explaining the extended x-ray absorption fine structure (EXAFS). This breakthrough led to development of XAFS spectroscopy as an analytical tool for determining the local molecular structure of condensed phases. Stern discussed the unique advantages of XAFS spectroscopy in determining short-range structure, valence, local symmetry, and angular deviation of atoms from collinearity. He also described nanosecond-scale time-dependent studies involving laser excitations of atoms in tandem with photon pulses inherent in synchrotron radiation.

Donald L. Sparks (University of Delaware) gave an overview of the importance of understanding the kinetics of reactions and processes in soils and sediments. Given the extreme complexity of such matrices, it is challenging to measure chemical kinetics separate from diffusion. Soil chemical reactions involving inorganic and organic contaminants often show an initially rapid rate followed by a slow approach to steady state, with hysteresis upon reversal. Very short, real-time, molecular-scale analyses are needed to determine chemical kinetics in isolation from transport, and elucidate reaction mechanisms that determine the fate of environmental contaminants.

One promising method for measuring short-term kinetics is Quick-EXAFS. Wolfgang Caliebe (NSLS) compared various Quick-EXAFS systems and showed Quick-EXAFS spectra collected at the NSLS on time scales of seconds. The rate-limiting component for Quick-EXAFS is typically the detector. The application of time-resolved *in-situ* EXAFS analysis was illustrated for battery materials by Mali Balasubramanian (Advanced Photon Source). These examples showed how quasi-equilibrium states of chemical reactions could be probed on times scales of three to 30 minutes. For example, XAFS spectra revealed how chromium ions migrate between octahedral and tetrahedral positions in Mn-oxide systems.

The precipitation, growth, and crystallization of naturally occurring iron oxides under various redox and pH conditions regulate the mobility of chemicals in the environment by, for example, adsorption and encapsulation. Sam Shaw (Oxford University) described *in-situ*, synchrotron-based time-resolved small-angle x-ray scattering (SAXS) and time-resolved x-ray diffraction (TRXRD) studies that followed the growth of poorly ordered iron oxyhydroxide (ferrihydrite) and its transformation into goethite and hematite, which are more stable crystalline endproducts. Rapid (seconds to minutes) precipitation and crystal growth of ferrihydrite was followed with SAXS using a stopped-flow reaction cell. Adsorption of chemical species such as phosphate on freshly precipitated mineral phases slows the overall crystal growth and retards growth on specific crystal faces. Based on activation energies calculated from TRXRD, ferrihydrite apparently transforms to goethite by dissolution and precipitation and to hematite by solid-state transformations.

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John Parise (Stony Brook University) and James D. Martin (North Carolina State University) gave additional examples of using TRXRD to follow chemical kinetics. Parise showed how this technique could be used to optimize the synthesis of a titanium silicate material to maximize its selectivity for cesium, an important radioactive contaminant in nuclear waste. Highly selective binding of this element by ion exchange was attributed to structural distortions in the material. By following total scattering from nanocrystalline materials over time, evolution of the pair-distribution functions for FeS minerals were also determined. Martin used TRXRD coupled with differential scanning calorimetry to study the nucleation and growth of $ZnCl_2$ from melts. The rate of crystal growth was inversely related to the initial temperature of the melt. For sodalite, templating was found to be critical to crystal growth, with isothermal crystallization only occurring if the melt reached a critical temperature below the melting temperature, which allowed proper ordering of the templating cations.

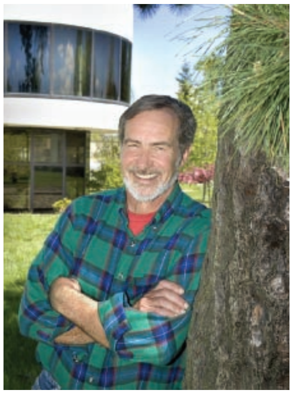
In summary, in-situ synchrotron-based x-ray absorption, scattering, and diffraction are successfully used to follow the kinetics and mechanisms of chemical processes in a variety of systems. Further developments and applications of these methods to probe reactions at increasingly shorter time scales in highly heterogeneous natural systems will advance our understanding of the short- and long-term fate of chemical contaminants in the environment.

— Dean Hesterberg and Jeff Fitts

Tony Lenhard Receives the 2005 UEC Community Service Award

May 24, 2005

This year, the NSLS Users' Executive Committee (UEC) presented the UEC Community Service Award to Tony Lenhard, Senior Technical Supervisor, NSLS User Science Division. The award is given for service, innovation, and dedication to NSLS users, and Tony is well deserving of that honor.



Tony Lenhard

As a tribute to nearly 25 years of dedicated support for NSLS experimenters and beamlines, members of the NSLS user community nominated Tony Lenhard for this year's award. Tony has demonstrated an uncommon dedication to improving the experimental setups at the NSLS. Here are quotes and paraphrased comments from some of the users who nominated Tony:

- Tony has supervised the Beamline Development and Support group that has been central to the construction, upgrade, and maintenance of all of the User Science Division beamlines: x-ray, ultraviolet, and infrared, including the insertion devices.

- As supervisor of that group, his superior traits have set the standard and inspired all of the NSLS User Science Division technical staff to excellence.

- "Tony is always ready to advise on the mechanical design of users' experimental apparatus, even under acute time pressure."

- "Tony is a treasure house of technical knowledge and skill in setting up beamlines and experiments. He has had a huge impact on the user community, based on his supervision of the technical work on every one of the NSLS-operated beamlines. Since many of the PRT beamlines look to the NSLS-constructed beamlines for technical guidance, Tony's mark is probably on every single beamline at the NSLS."

NSLS UEC Vice Chair Peter Stephens presents the 2005 UEC Community Service Award to Tony Lenhard at the Users' Meeting banquet.

- Many of the people who wrote in support of his nomination made particular mention of his special talent for coming up with a simple solution to a mechanical problem, often quite different from what the experimenter initially visualized.

- "I don't believe the community properly appreciates his contributions to the many experiments that otherwise would have been delayed."

- Tony is patient and unflappable to a remarkable degree, especially considering the excited state that experimenters are often in when facing technical difficulties at the start of an experiment.

- "Tony may be one of the most underappreciated treasures in the NSLS. He is often on the spot when users arrive and he has to make some quick modifications to equipment to allow them to complete an experiment."

- "Tony plays a crucial role in assisting NSLS users while they are performing experiments. Every user facility needs a person like Tony: someone who knows how to build or modify any mechanical part in a short amount of time, and someone who is kind enough to help guide experimenters in the right direction."

- Tony has served the NSLS User Community in the ways described above for essentially the entire life of the NSLS. Ironically, many of the users whose experiments run well have never even met him. It is time for him to be recognized for this service via this Community Service Award.

Peter Stephens vice chair of the UEC, presented the award to Tony at the NSLS Users' Meeting banquet on the evening of Tuesday, May 24th. Tony received a $250 gift certificate and his name was engraved on the plaque on display in the NSLS lobby. Congratulations Tony, and thanks for a job well done!

— Peter Stephens